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Electro Spray Method for Flexible Display

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14. ABSTRACT
The goal of this research is to explore methods of developing aerosol-based printable thin-film transistors (TFTs) from zinc oxide (ZnO). This technique would vastly simplify high performance TFT fabrication with the potential to make low-cost printable electronic and opto-electronic devices compatible with transparent, flexible electronics. If successful, Aerosol-based methods of fabricating high-mobility ZnO transistors would aid in creating high performance displays.

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Abstract:

InZnO TFTs fabricated from an aqueous solution exhibited a high field effect mobility of 19.5 $cm^2/V \cdot s$ and a low off-current of 10^{-13} A at 300 ºC. Carbon-related defects were considered acting as trap centers in InZnO thin films and affected on- and off-currents of TFTs. Since much lower concentrations of carbon remained in the aqueous solution-derived InZnO thin films, i.e., the capture of electrons significantly reduced, much higher on- and lower off-current were obtained comparing with organic solution-derived TFTs which were fabricated at much higher temperatures of 600 and 700 ºC.

Introduction:

Zinc oxide (ZnO)-based materials have attracted significant attention for the application of emerging electronic devices including thin film transistor (TFT) backplanes for flexible displays or transparent active matrix organic light-emitting diode (AMOLED) due to their excellent optical and high mobility.[1-6] For the fabrication of ZnO-based thin films, vacuum processes, such as, sputtering, pulsed laser deposition and plasma enhanced chemical vapor deposition were preferred.[1-6] Vacuum-deposition methods require expensive equipment and result in high manufacturing costs. As an alternative technique, the solution-process deposition method, is very likely to be used in the future mass-production of thin film oxides because of its low cost, simplicity, high

throughput, and accurate control of composition for multi-component thin films.[2-7] However, high annealing temperatures (400 °C or more) are commonly required to decompose the organic additives as well as the crystallization of the semiconducting oxides to obtain high property TFTs by the use of organic solutions.[2-7]

Banger *et al.* adopted a "sol-gel on chip" method and fabricated high mobility of about 12 $cm^2/(V\cdot s)$ InZnO (IZO) TFTs at 250 ºC.[8)] However, fabrication steps requiring anhydrous conditions which expensive and complicated.[8-9)] Kim *et al.* reported the fabrication of IZO thin films via combustion processing and obtained mobility values of 3.20 and 9.78 $cm^2/(V\cdot s)$ when the IZO thin films were annealed at 300 and 400 ºC.[10)] In Kim's report, they used acetylacetone or urea as a fuel and metal nitrates as metal sources in solutions. Through the high self-generated energies by the combustion of acetylacetone or urea in solution, converting of precursors into corresponding oxides only needed a simple process and low process temperatures. However, IZO TFTs with mobility higher than 10 $cm^2/(V\cdot s)$ using a simple process at temperatures not higher than 300 ºC is rarely reported.

Remained organic parts are considered as a large barrier to increase the mobility of solution-process-derived TFTs. Therefore, we used H_2O as the solvent in our precursor solution. The use of H_2O could decrease the introduction of organic impurities. Moreover, the H_2O is a better oxidizer than O_2. The binding energy of O-O in O_2 is 5.1 eV, and the O-H (O-OH) in H_2O is 4.8 eV. The O derived from H_2O was expected to be more effective for the generation of metal-oxide-metal matrix. It was also discussed by Nomura *et al.* that the O derived from H_2O was much mobile than those from the O_2.[10)] Deposited thin film was heated on hot plates at 150 ºC for 5 min and 300 ºC for 1 h after the solution was spin-coated on substrate. Thickness of the fabricated IZO thin film was about 10 nm. The thin film will be abbreviated as A-IZO300. In order to understand the effect of this novel solution (A-IZO solution), TFTs with IZO thin films derived by a common metal organic decomposition (MOD) solution (Kojundo Chemical Lab. Co., Ltd.) were also fabricated. Annealing temperatures of 600 and 700 ºC were used to fabricate MOD solution-deposited IZO thin films. These IZO thin films will be abbreviated as IZO600 and IZO700. Thicknesses of the MOD solution-derived IZO thin films were about 20 nm. A bottom-gated structure for TFT fabrications was adopted. Heavily doped p-type silicon wafers were used as gate electrodes. Thermally grown SiO_2 thin films were used as gate dielectrics. Pt(10 nm)/Ti(90 nm) films were deposited using electron-beam evaporation method and were patterned via a typical lift-off process to form source and drain electrodes on IZO channel layers. Channel width and length values of these TFTs were about 500 and 50 μm, respectively.

Thermal behaviors of A-IZO and MOD solutions were monitored under air atmosphere from room temperature to 500 °C using a thermogravimetry-differential thermal analysis (TG-DTA; EXSTAR TG/DTA6000), as shown in Fig. 1. An exothermic peak around 203 °C with no significant weight loss was observed. However, a much higher temperature of 348 °C needed to be used for completing the pyrolysis reaction of the MOD solution. Unlike the MOD solution, which exhibit broad endotherms for organic solvent removal and exotherms for oxide lattice formation, the A-IZO solution, exhibit a single intense exothermic peak in the DTA that corresponds exactly to the abrupt mass loss in the TG and is sufficient to drive the reaction rapidly to completion.

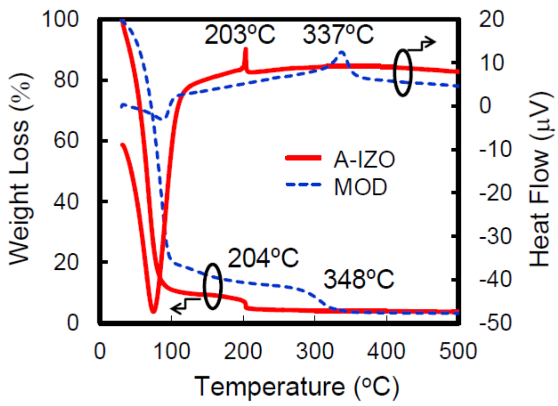

Figure 1 TG-DTA result of A-IZO and MOD solutions.

Output and transfer characteristics of the A-IZO300 TFT are depicted in Fig. 2(a) and 2(b), respectively. Good pinch off and saturation states were obtained, as shown in the output diagram (Fig. 2(a)). The saturation state of the TFT was observed at drain voltages (V_{ds}) of lower than 5 V. Various parameters were calculated at V_{ds} of 5 V. The values of μ_{FET}, on/off current ratio and subthreshold swing (S) were 19.5 cm^2/V· s, exceeding 10^9, and 0.36 V/decade, respectively. Low off-currents of 10^{-13} A were also obtained, which were lower than many solution process-derived IZO TFTs.[2-6)] No higher μ_{FET} value was reported by the use of a solution process at 300 °C with such a low off-current until now.

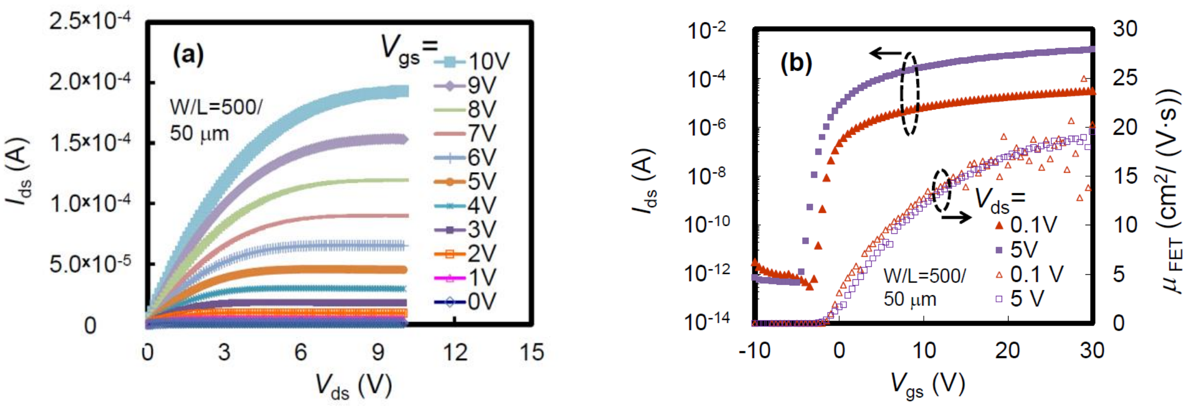

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Figure 2 (a) Output and (b) transfer characteristics of the A-IZO300 TFT.

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IZO600, IZO700 and A-IZO300 TFTs are compared in Fig. 3. Among these TFTs, the A-IZO300 TFT showed the lowest off-current and the highest on-current. The IZO700 TFT showed a higher on-current and a lower off-current than those of the IZO600 TFT. The μ_{FET} of the IZO600 and IZO700 TFT were about 0.1 and 2.2 $cm^2/(V \cdot s)$, respectively. They were much lower than that of the A-IZO300 TFT even though much higher annealing temperatures were carried out on the fabrication of IZO600 and IZO700 thin films.

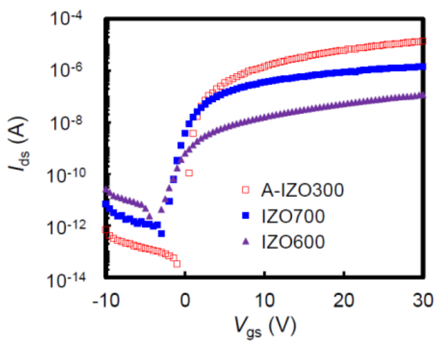

Figure 3 Transfer characteristics (measured at V_{ds}=0.1 V) of A-IZO300, IZO600 and IZO700 TFTs.

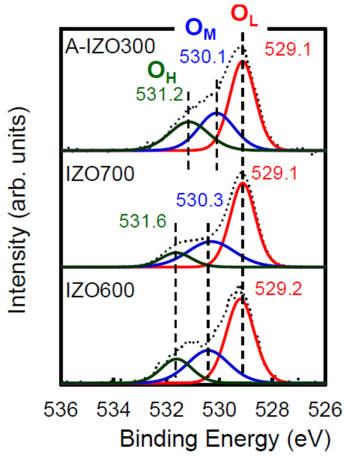

Figure 4 Binding energies of O 1s of A-IZO300, IZO600 and IZO700 thin films. O_L are attributed to stoichiometric oxidized metal bonds. O_M is associated with oxygen-deficient regions within the IZO compound. O_H is due to the loosely bonding of O with carbon (C) or hydrogen.

It is well known that characteristics of oxide semiconductor TFTs intensively affected by oxygen vacancies in channel layers. Binding energies of the O 1s in IZO thin films were investigated by X-ray Photoelectron Spectroscopy (XPS; KRATOS AXIS-165) using a mono-Al Kα radiation, as shown in Fig. 4. The O 1s peaks were divided into three peaks that were determined by Gaussian fitting. Fitted peaks with low binding energies (O_L) are attributed to stoichiometric oxidized metal bonds (O-M).[11-13] Binding energies of O_L were 529.2, 529.1 and 529.1 eV for IZO600, IZO700 and A-IZO300 thin films, respectively. It suggested that the A-IZO300 thin film was well fabricated even though it

was annealed at a very low temperature of 300 ºC. The medium binding energy (O_M) component, around 530 eV, is associated with oxygen-deficient regions within the IZO compound.[11-13] The highest binding-energy peak (O_H) is due to the loosely bonding of O with carbon (C) or hydrogen.[11-13] The $O_M/(O_H+O_M+O_L)$ ratio represents the relative amounts of oxygen vacancies in thin films, which were about 30%, 32% and 28% for IZO600, IZO700 and A-IZO300 thin films, respectively. Most papers suggested that larger amount of oxygen vacancies could provide higher on and off-currents.[14-16] In this work, although the largest amount of oxygen vacancies was in the IZO700, its on-current was lower than that of the A-IZO300 TFT and the off-current was lower than that of the IZO-600 TFT. Therefore, the on and off-currents were not only decided by oxygen vacancies in IZO thin films.

Impurity of C in IZO thin films were investigated by secondary ion-microprobe mass spectrometry (SIMS; Ion-Microprobe Atomika-6500) using a Cs ion beam, as shown in Fig. 5. The concentration of C in MOD solution derived thin films decreased as annealing temperature increased from 600 to 700 ºC. Remarkably lower concentration of C was found in the A-IZO300 thin film than in MOD solution-derived thin films even though the annealing temperature was as low as 300 ºC. It was found in the XPS result that the binding energy of the O_H in the A-IZO300 thin film shifted to a lower energy than those in the IZO600 and IZO700 thin films. Based on the SIMS result, we suggested that the binding energy shift of the O_H was due to the significantly decreased C impurity.

It is not the same as vacuum process-derived TFTs, organic remains significantly affect TFTs' properties of solution process-derived oxide TFTs, which was introduced in the beginning of this report. However, rare reports discussed the effect of C on TFTs' properties in detail. We found in this research that the variation of C concentrations fitted the transfer characteristics very well. In the IZO600 film, largest amount of C was observed, the IZO600 TFT showed the lowest on-current and the highest off-current. On the contrary, in the A-IZO300 film, least amount of C was found, the A-IZO TFT showed the highest on-current and the lowest off-current. Therefore, we suggested that the C-related impurities, such as C^{4+} and $(CO)^{2+}$, in IZO thin films acted as trap centers of electrons and significantly affected on and off-currents of TFTs. The schematic diagram of the C-related trap centers affected on and off-currents of IZO TFTs is shown in Fig. 6. These trap centers captured electrons which were generated by oxygen vacancies in channel thin films when TFTs act on on-states, consequently prevented the moving of carriers. When TFTs act on off-states, the channel layers were in depletion states, high electric fields were applied on IZO thin films. These high electric fields induced the release of captured electrons from the trap centers, consequently contributed off-currents.

In the A-IZO300 thin film, the lowest amount of C remained, thus the lowest amount of electrons was captured. Therefore, the highest on-current was obtained even though least amount of oxygen vacancies was observed. In the off-state, since the lowest amount of electrons was released from the C-related trap centers in the A-IZO300 thin film, it showed the lowest off-current. According to many other researchers' reports, solution-process derived oxide semiconductor TFTs commonly showed higher off-current than vacuum process-derived TFTs.[2-6] It might also due to remained C in oxide semiconductors.

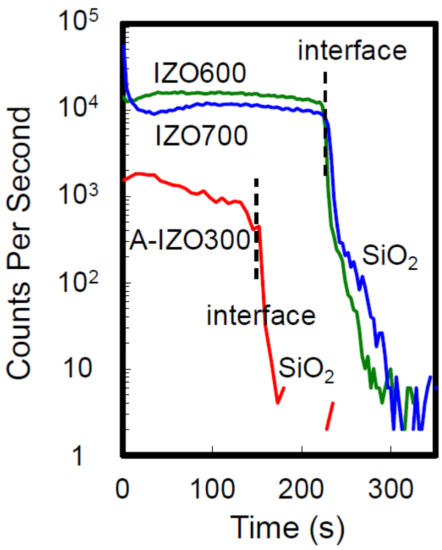

Figure 5 Depth profiles of C in A-IZO300, IZO600 and IZO700 thin films.

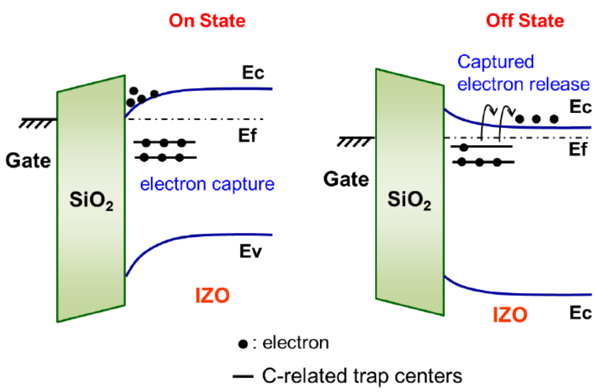

Figure 6 Schematic diagram of the C affected on and off-currents of IZO TFTs. The energy levels of C-related defects might be around Ec-0.5 eV.

Gate bias stress instability of the A-IZO300 TFT was also investigated by applying a positive gate voltage (V_{gs}) of 20 V on the TFT from 1 to 1000 s. In order to elucidate the effect of environment on the gate bias stress instability,[18-20] the A-IZO300 TFT was passivated by an atomic layer deposition derived Al_2O_3 thin film with the thickness of 50 nm. The threshold voltage shift(ΔV_{th}) of unpassivated and passivated TFTs are shown in Fig. 7. Here, the V_{th} value was extracted from linear fittings to the plot of the square root of drain current (I_{ds}) versus V_{gs}. After the passivation, a much smaller ΔV_{th} (after 1000 s) of 1.9 V was obtained comparing with the unpassivated TFT which was 3.4 V.

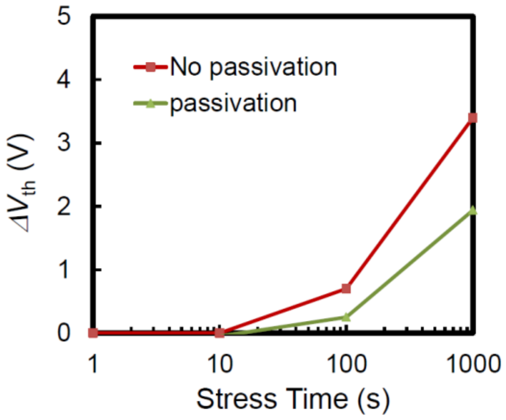

Figure 7 Threshold voltage shift (ΔV_{th}) of unpassivated and passivated A-IZO300 TFTs.

In conclusion, we fabricated IZO thin films from an aqueous solution containing metal sources. Very high μ_{FTE} and on/off current ratio of 19.5 $cm^2/V \cdot s$ and exceeding 10^9, respectively, were obtained for the fabricated IZO TFT even though the annealing temperature was as low as 300 ºC. Low off-current of 10^{-13} A was also obtained. We proposed that the C existed in IZO thin films and acted as trap centers to capture carriers in channels. The trapped carriers were released while TFTs acted on off-states. Since much lower concentration of C impurities remained in the IZO thin film fabricated by this solution, much higher on-current and lower off-current were obtained than MOD solution-derived IZO TFTs which were fabricated by much higher temperatures of 600 and 700 ºC. On the other hand, a good gate bias instability with the ΔV_{th} of 1.9 V was obtained after the passivation of an atomic layer deposition derived Al_2O_3 thin film.

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Achievement

Award

IEEE/IMFEDK2016 Student Paper Award

1) I.Raifuku and Y.Uraoka et al,
   “Potential of Perovskite Solar Cells for Power Sources of IoT Applications”

IEEE/AMFPD2015 Student Paper Award

2) Kahori Kise and Y.Uraoka et al,
   7-3 “Analysis of Self-Heating Phenomenon in Oxide Thin-Film Transistors under Pulsed Bias Voltage”

IEEE/AMFPD2015 ECS Japan Section Young Researcher Award

1) M.Horita and Y.Uraoka et al,
6-3 "Unseeded Growth of Poly-Crystalline Ge with (111) Surface Orientation on Insulator by Pulsed Green Laser Annealing"

IDW '15 Best Poster Award

1) C. Kulchaisit and Y.Uraoka et al**,**
AMDp1-16L **"**Reliability Improvement of Amorphous InGaZnO Thin-Film Transistors by Less Hydroxyl-Groups Siloxane Passivation"
2) H. Yamazaki and Y.Uraoka et al,
FLXp2-3, "Thermal Analysis of Oxide Thin Film Transistor with Fluorinated Silicon Nitride Gate Insulator".
3) M.Ochi and Y.Uraoka et al,
AMDp1-3, "Behavior of a-IGZTO TFTs with BCE Structures Containing Floating Metal Electrodes"

<u>Invited Talk</u>

1. Y.Uraoka, International Thin Film Transistor Workshop, "Oxide Thin Film Transistors for Flexible Device", Rennes, France, February 26-27, 2015.
2. Y.Uraoka, The 11^{th} Pacific Rim Conference of Ceramic Societies, "Oxide Thin Film Transistors for Flexible Displays", August 30 to September 4, 2015, ICC Jeju, Jeju, Korea.
3. Y.Uraoka, The 15^{th} International Meeting on Information Display, "Oxide Thin Film Transistors for Flexible Device", August 18-21, 2015, Exco, Daegue, Korea.
4. Y.Uraoka, J.P. Bermundo, M.Fujii, Y.Ishikawa, "Oxide Thin Film Transistors for Flexible Devices", IWFPE 2015, Korea, 2015
5. Y.Uraoka, The 16^{th} International Meeting on Information Display, "Oxide Thin Film Transistors for Flexible Device", August 23-25, 2016, ICC Cheju, Korea.

<u>Journal</u>

1) Juan Paolo Bermundo, Yasuaki Ishikawa, Mami N. Fujii, Michel van der Zwan, Toshiaki Nonaka, Ryoichi Ishihara, Hiroshi Ikenoue, Yukiharu Uraoka, "Low Temperature Excimer Laser Annealing of a-InGaZnO Thin-Film Transistors

Passivated by Organic Hybrid Passivation Layer”, Applied Physics Letter, 2015.

2) M.N.Fujii, Y.Ishikawa, K.Miwa, H.Okada, Y.Uraoka, S.Ono, “High-density carrier-accumulated and electrically stable oxide thin-film transistors from ion-gel gate dielectric”, Scientific Reports, 5:18168, 2015.
3) C. Kulchaisit, Y.Ishikawa, M. Fujii, Y.Uraoka, “Reliability Improvement of Amorphous InGaZnO Thin-Film Transistors by Less-Hydroxyl-Groups Siloxane Passivation”, Journal of Display Technology, 12(3), 1-1, 2015.
4) J. P. Bermundo, Y.Ishikawa, M.N.Fujii, T.Nonaka, R. Ishikawa, H. Ikenoue and Y.Uraoka, “Effect of excimer laser annealing on a-IngaZnO thin-film transistors passivated by solution-processed hybrid passivation layers”, J. Phys. D. Appl. Phys. 49(2016) 035102 (7pp).
5) Kahori Kise, Y.Ishikawa, Y.Uraoka, “Self-heating induced instability of oxide thin film transistors under dynamic stress”, Appl. Phys. Lett., 108, 02501 (2016).

International Conference

1) C. Kulchaisit and Y.Uraoka et al,
AMDp1-16L “Reliability Improvement of Amorphous InGaZnO Thin-Film Transistors by Less Hydroxyl-Groups Siloxane Passivation”, International Display workshop 2015 (IDW’15), 2015.
2) H. Yamazaki and Y.Uraoka et al,
FLXp2-3, “Thermal Analysis of Oxide Thin Film Transistor with Fluorinated Silicon Nitride Gate Insulator”, International Display workshop 2015 (IDW’15), 2015.
3) M.Ochi and Y.Uraoka et al,
AMDp1-3, “Behavior of a-IGZTO TFTs with BCE Structures Containing Floating Metal Electrodes”, International Display workshop 2015 (IDW’15), 2015.
4) M.Fujii and Y.Uraoka et al, “High-performance InGaZnO Thin Film Transistor Ionic Liquid Gate Dielectric”, International Thin Film Transistor Conference (ITC), 2015.
5) D.Hishitani and Y.Uraoka et al, “Polycrystalline Silicon Thin Film Transistors with Solution Derived SiO2 Gate Insulator Formed Using CO2 Laser Annealing”, International Thin Film Transistor Conference (ITC), 2015.
6) J.P.Bermundo et al, “XeCl and KrF Excimer Laser Annealing of Oxide Thin-Film Transistors with Hybrid Passivation Layer”, International Thin Film Transistor Conference (ITC), 2015.
7) C.Kulchaisit and Y.Uraoka et al, “Improvement of the Reliability of Bottom Gate

Amorphous InGaZnO Thin-Film Transistors with Siloxane Layer", International Thin Film Transistor Conference (ITC), 2015.
8) H.Yamazaki and Y.Uraoka et al, "Oxide Thin Film Transistors for Flexible Devices", International Thin Film Transistor Conference (ITC), 2015.

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